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B. S. El-Dasher, S. G. Torres

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The Effect of Solution Annealing on Alloy 22 Weld Properties

Bassem S. El-Dasher and Sharon G. Torres
*Lawrence Livermore National Laboratory, L-631
Livermore, CA 94551*

Abstract - The effect of solution annealing temperature on the microstructure and observed corrosion attack mode in Alloy 22 welds was assessed. Specimens were examined in the as-welded state as well as solution annealed for 20 minutes at temperatures ranging from 1075°C to 1300°C. The microstructures of the specimens were first mapped using electron backscatter diffraction to determine the grain structure evolution due to solution annealing. Full recrystallization of the fusion zone was only observed in the 1200°C and 1300°C specimens, although the 1300°C specimen showed abnormal grain growth. As-welded, 1121°C and 1200°C specimens were also subjected to electrochemical testing in a 6 molal NaCl + 0.9 molal KNO₃ environment to initiate crevice corrosion. Examination of the specimen surfaces after corrosion testing showed that in the as-welded specimen, corrosion was present in both the weld dendrites as well as around the secondary phases. However, the specimen solution annealed at 1121°C showed corrosion only at secondary phases and the specimen annealed at 1200°C showed pitting corrosion only in a handful of grains.

I. INTRODUCTION

Alloy 22 (UNS N06022) is a nickel-based alloy containing approximately 22% chromium, 13% molybdenum, 3% iron, and 3% tungsten, and is the candidate material for the outer barrier of the nuclear waste containers for the proposed repository site at Yucca Mountain, Nevada. Its selection is primarily due to its excellent resistance to pitting and crevice corrosion, as well as to stress corrosion cracking^[1,2]. However, it is known that Alloy 22 welds form potentially detrimental intermetallic tetrahedrally (or topologically) close packed (TCP) phases upon solidification^[3]. The effect of these phases on material properties was highlighted in a study of aged Alloy 22 specimens, which correlated a decrease in corrosion performance and mechanical properties to TCP phase formation^[4]. The implications of this are clear when one considers that multiple welds are required for the construction of the outer barrier.

Traditionally solution annealing has been employed to eliminate the TCP phases from solidified microstructures, and is performed at the manufacturer recommended temperature of 1121°C (2050°F). Two studies on the effect of solution annealing on TCP phase stability have been performed to date. In the first study, specimens were treated at temperatures ranging from 1125°C to 1300°C^[5]. It was determined that the TCP phases did not dissolve at 1125°C for treatments up to 1

hour, or at 1200°C and 1300°C after 15 minutes. Volume fraction measurements indicated that the TCP phases grew during the solution annealing. In contrast, the second study^[6] compared a specimen solution annealed at 1121°C for 20 minutes to an as-welded specimen, and showed a relative TCP phase volume fraction decrease of approximately 5% after solution annealing.

In this work we examine the behavior of TCP phase stability and microstructural evolution of Alloy 22 welds solution annealed as a function of solution annealing temperature (1075°C to 1300°C). We also examine the corrosion behavior of the specimens solution annealed for 20 minutes at 1075°C and 1121°C and compare it to the corrosion behavior of the as-welded specimen.

II. EXPERIMENTAL

Specimens used in this study were cut from a 30" (762mm) long, 1.5" (38.1mm) thick welded plates. The plates were produced by double-U multi-pass gas-tungsten-arc welds fabricated using 1.5" mill annealed Alloy 22 plates and matching Alloy 22 filler wire (ERNiCrMo-10).

Five specimens were prepared for microstructural characterization. Four specimens were annealed at 1075°C, 1121°C, 1200°C, and 1300°C in preheated air furnaces for 20 minutes. The specimens were then

metallographically prepared, without etching. Two methods were applied to characterize the weld microstructure: backscatter electron imaging in a scanning electron microscope (SEM) to characterize the TCP phases and electron backscatter diffraction (EBSD) to determine the weld microstructure.

For corrosion testing, nine specimens were produced with three each in the as-welded condition, solution annealed at 1121°C and solution annealed at 1200°C. All anneals were performed at the respective temperature for 20 minutes. The specimens were cut such that they could be setup for corrosion testing as a prism crevice assembly (PCA). The set-up is based on crevice formers described by the American Society for Testing and Materials (ASTM) in ASTM G 48 and G61.

Electrochemical tests were conducted on the PCA's in an environment of 6 molal NaCl + 0.9 molal KNO₃ at a temperature of 90°C. These consisted of cyclic potentiodynamic polarization (CP) tests starting at approximately 100 mV below the corrosion potential (determined from previous open circuit potential tests), and continuing until a current density of 1 mAcm⁻² was reached before the scan was reversed.

III. RESULTS

Figure 1 shows the microstructure of the specimens solution annealed for 20 minutes at the various temperatures in comparison to the as-welded specimen. Recrystallization occurs at substantially different rates for the different temperatures, and abnormal grain growth occurs at 1300°C. Figure 1(f) shows the grain size distribution, in which it is apparent that the most homogenous microstructure is that of the specimen solution annealed at 1200°C. Analysis of the specimens annealed for longer durations (not shown here) show that the TCP phase dissolution can be achieved by solution annealing for 24 hours at either 1075°C or 1121°C, or 20 minutes at either 1200°C or 1300°C. It was also seen that full recrystallization of Alloy 22 welds can be achieved by solution annealing for 168 hours at 1075°C, 24 hours at 1121°C, and 20 minutes at 1200°C.

In Figure 2, the cyclic polarization curves for the as-welded, 1121°C for 20 minutes, and 1200°C for 20 minutes specimens are shown. Note that the study was limited to these three types of specimens since the microstructure of the 1075°C specimen is too similar to the as-welded specimen, and that abnormal grain growth was observed in the 1300°C specimen. From Figure 2 it is clear that the cyclic polarization curves for the three types of microstructures are different.

Although the electrochemical studies show different behavior between the three types of specimens, it is difficult to accurately determine the exact nature of these behaviors without more detailed and sensitive studies. However what is apparent from the tests is that the breakdown potentials decrease, and the repassivation potentials increase, with increasing solution annealing temperatures. The values of the breakdown potential (E_{20}) and the repassivation potential (E_{r1}) are listed in Table I for the nine specimens studied. The crossover potential is not reported as the 1200°C specimens showed no hysteresis.

Interestingly, the cyclic polarization behavior observed corresponds to the corrosion behavior as seen in Figure 3. For the as-welded specimen, the majority of the corrosion is seen in the weld dendrites (Figure 3(a)). This behavior is expected, as solute segregation during solidification has been known to cause increased amounts of Cr and Mo in the interdendritic region. Also, since TCP phases are rich in Cr and Mo, their presence in turn requires the regions around them to be depleted from these elements.

After annealing at 1121°C for 20 minutes, it is expected that along with recrystallization, the Cr and Mo distribution becomes more homogeneous. In Figure 3(c) and (d), it can be seen that material dissolution occurs in the interdendritic regions, specifically at the areas corresponding to TCP phase locations. In the higher magnification image (Figure 3(d)), the microstructure shows TCP phase shaped pits. It appears that since virtually all the corrosion occurred at the TCP phase/matrix interfaces, the TCP phases fell out of the specimen during the corrosion test. This then indicates that solution annealing at 1121°C for 20 minutes minimizes the likelihood of dendritic corrosion, at the expense of focusing corrosion around the TCP phases.

Post-corrosion characterization of the specimen annealed at 1200°C for 20 minutes was somewhat difficult, as only one small area under the crevice showed signs of corrosion. Micrographs for this area are presented in Figures 3(e) and (f), and it can be seen that the corrosion mode is typical of crevice corrosion in wrought materials, with many fine pits seen in multiple grains. Overall, it is apparent that the specimen annealed at 1200°C had less localized corrosion at the micro-level than the other specimens.

IV. CONCLUSIONS

Specimens of Alloy 22 multi-pass gas-tungsten-arc-welds were solution annealed at various temperatures and

for various durations. Microstructural characterization and corrosion testing revealed that:

- Full recrystallization of Alloy 22 welds can be achieved by solution annealing for 168 hours at 1075°C, 24 hours at 1121°C, and 20 minutes at 1200°C. At 1300°C, annealing for 20 minutes produces a microstructure with a abnormal grain sizes in the fusion zone, indicating a significant driving force for grain growth and coarsening.
- Of the fully recrystallized specimens observed, the specimen annealed at 1200°C for 20 minutes yielded the most homogeneous grains.
- Cyclic polarization tests showed differences between the corrosion behavior of the three types of specimens, and these differences seemed to correspond to differing modes of corrosive attack at the micro-level.
- The as-welded specimens were primarily attacked in the secondary dendrites, and around the secondary phases in the interdendritic region, whereas the specimen annealed at 1121°C showed attack solely around the TCP phases in the interdendritic regions.
- The specimen annealed at 1200°C, which was fully recrystallized, showed pitting in only a few grains, with no apparent pattern or preference (similar to wrought material).

ACKNOWLEDGMENTS

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TABLE I- Critical potentials determined from CPP tests on Alloy 22 as-welded specimens, and specimens solution annealed at 1121°C and 1200°C for 20minutes.

Specimen	Breakdown Potential (E₂₀, mV)	Repassivation Potential (E_{r1},mV)
As-welded		
1	597	-31
2	475	-73
3	545	-62
Solution Annealed at 1121°C for 20 minutes		
1	457	-78
2	132	-77
3	107	-27
Solution Annealed at 1200°C for 20 minutes		
1	91	348
2	55	-10
3	103	466

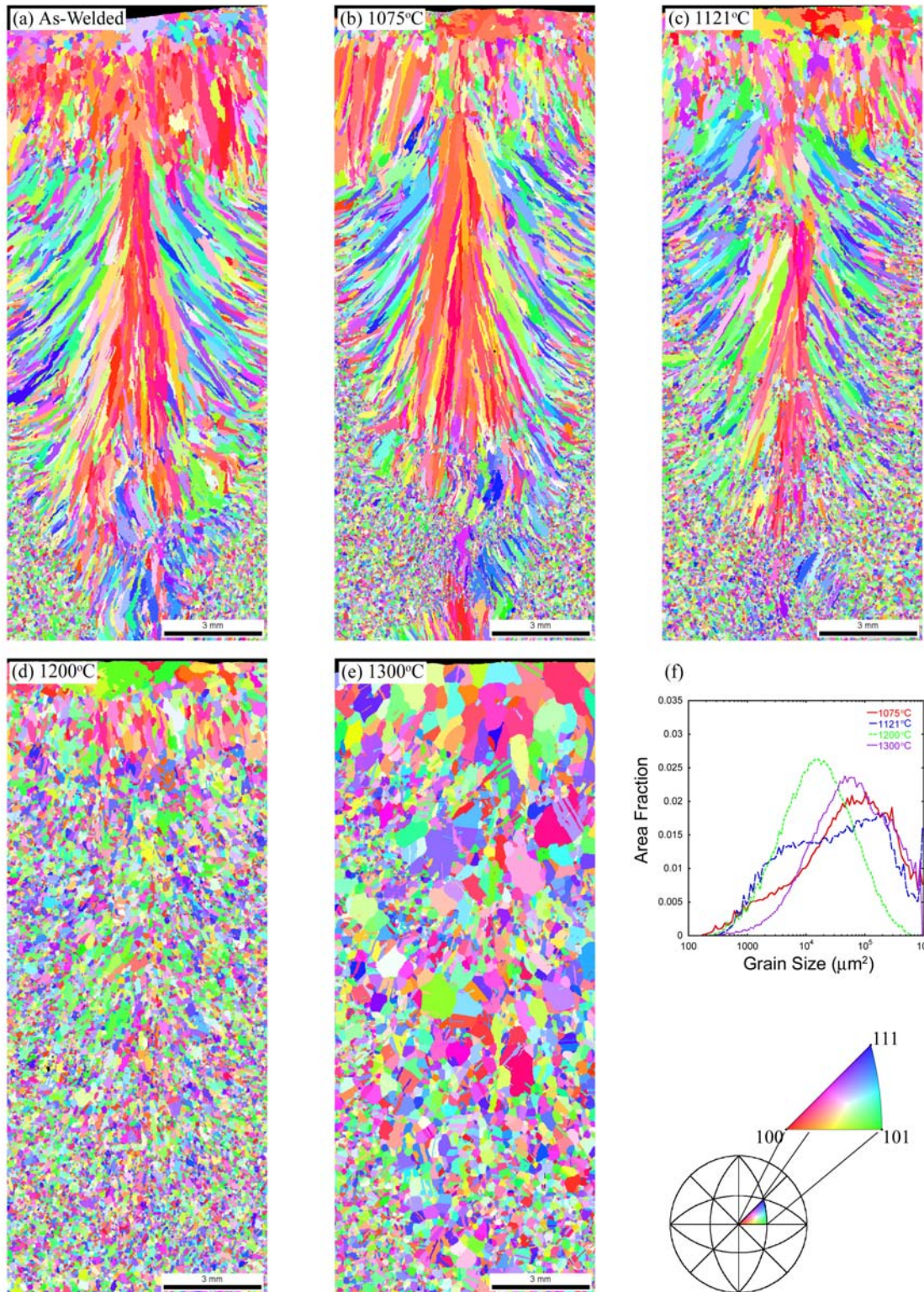


Figure 1- Weld microstructure the (a) as-welded specimens, and (b) through (e) the specimens solution annealed for 20 minutes. Grain size distribution of the specimens solution annealed for 20 minutes is shown in (f).

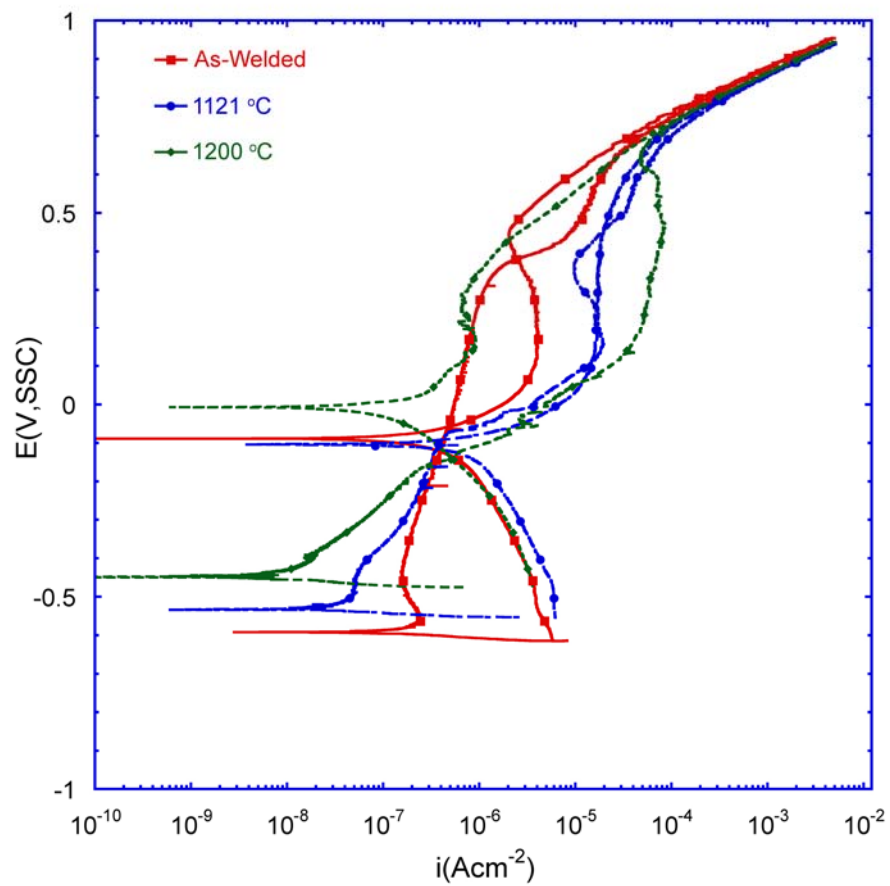


Figure 2- Cyclic Polarization curves for the as-welded and solution annealed specimens in 6m NaCl + 0.9m KNO₃ at 90°C.

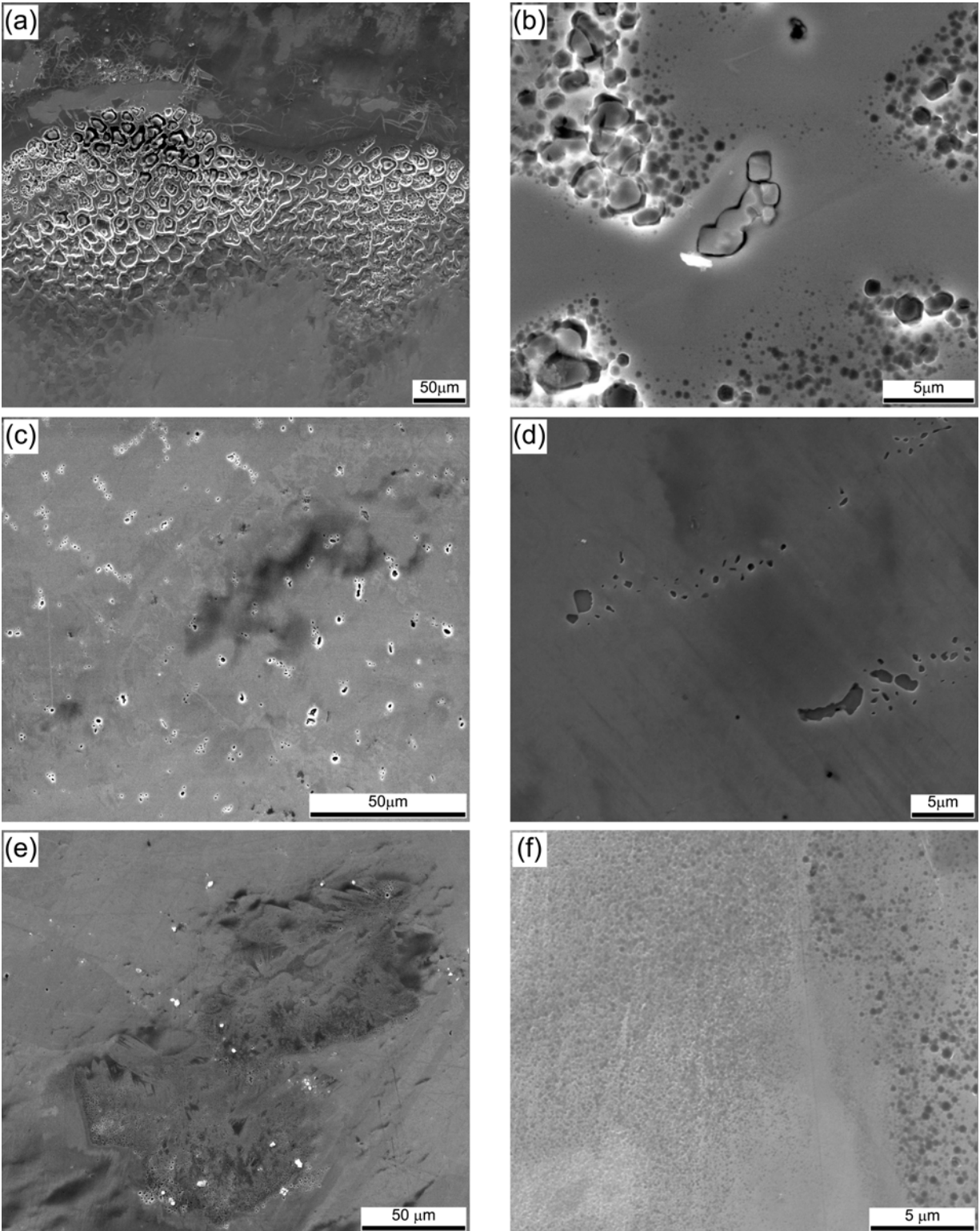


Figure 3- SEM Micrographs showing the areas of observed corrosion for: (a) and (b) the as-welded specimen, (c) and (d) the specimen solution annealed for 20 minutes at 1121°C, and (e) and (f) the specimen solution annealed for 20 minutes at 1200°C.